



Research Article

Extraction and Characterization of Oil from Watermelon Seed

UP Ojukwu and Ugwu OC

Department of Polymer and Textile Engineering, Nnamdi Azikiwe University Awka

Department of Production Technology, Nnamdi Azikiwe University, Awka

*Corresponding author: ujupauline3@yahoo.com

Article History: 12337 Received: 21/3/21 Revised: 22/6/21 Accepted: 2/7/21

ABSTRACT

Oil was extracted from dried and powdered water melon (*Citrullus lanatus*) seed using soxhlet extraction method with n-hexane (40-60°C) as the solvent. The oil yield was 40%. Characterization of the oil showed that it had acid value of 2.4mg/g, iodine value of 1.47208mg/g and saponification value of 688mg/g. The iodine value indicated that it was a non-drying oil and the high saponification value showed that the oil would be good for making hair shampoo, soap and creams. Hair shampoo of commercial quality was formulated from the oil.

Key words: Watermelon seed, Soxhlet extraction.

INTRODUCTION

Fats and oil are esters of fatty acids from trihydric alcohols and glycerol and are referred to as fatty glyceryl esters. Animal and vegetable fats are mainly composed of triglycerides of fatty acid such as stearic, palmitic and oleic, a molecule of such a triglyceride being derived by the combination of one molecule of glyceride and three fatty acid molecules (Morrison and Boyd, 1997).

Fat differs from oil in that it is solid at room temperature while oil is a liquid at the same condition. Another difference is that fats are mainly glyceryl esters of unsaturated fatty acids while oils are not.

Oils are liquid natural substances extracted from soils and tissues of plants and animals. They are viscous combustible liquids that are soluble in certain organic solvents like hexane, benzene, ether and chloroform. They are insoluble in water and slightly soluble in alcohol except castor oil which is readily soluble in alcohol.

Removal of oil from seeds are done by two physical processes namely solution extraction and diffusion extraction in plants. Due to high steam and labour requirements such plants are being steadily replaced with continuous solvent extraction plants whose main component is the extractor which can be the immersion percolation or direct extraction type. The combined use of these two types of extractors makes it possible to extract oil with no need for continuous screw presses expellers (Ernesto, 1976).

Solvent extraction which is also known as liquid liquid extraction is affected by treating a mixture of different

substances with a selective liquid solvent. At least one of the components of the mixture must be immiscible or partly miscible with the treating solvent so that at least two phases can be formed over the entire range of operating condition. The effectiveness of separation is increased by the use of a given amount of extractive solvent in successively smaller proportion rather than by a single extraction with the total amount (Van, 1976). Other forms of solvent extraction include leaching, washing and precipitate extraction (salting out). Removal of oil from the bearing materials of plants or origin is also done by subjecting the material to great pressure in hydraulic press. Essential oils from saps and tissues of plant are extracted by expression, adsorption on purified fat (effleurage) and steam distillation. The kinetics of solvent extraction of oils from seeds have been studied by various authors (Nwafor, 1995).

The characterization of oils and fat is needed for an assessment of quality and purity as well as for their identification. A number of chemical and physical constants are necessary. The physical constants include melting point, viscosity, specific gravity, refractive index, colour and solubility. The chemical constants are saponification value, free fatty acid value and iodine value (Azogu, 2010).

The application of fats and oil in industries depends on their physicochemical constants such as iodine value, acid value and saponification value. These values show their chemical and physical characteristics (Ibemesi, 1992). Extraction of oil from the seed of a local plant, watermelon is done so as to obtain the quality and possible use in industrial processes.

MATERIALS AND METHODS

Sample Preparation

The water melon seed was obtained from Awka, Anambra State of Nigeria. The seeds were removed from the seed coat and dried very well under the sun for several days. The dried seeds were ground to powder and stored in black polythene bag ready for analysis.

Extraction of Oil

Solvent extraction method was used to extract oil from the water melon seed. The ground sample was poured into a thimble and dropped into soxhlet extractor which was fitted to a 250 ml round bottom flask. Boiling chips were put into the round bottom flask to prevent bumping during heating and enough quantity of n-hexane (b.p 40-60°C) was poured into extractor containing the sample.

The extractor was coupled to a reflux condenser clamped to a retort stand. Extraction was done on a heating mantle. As the solvent was heated, its vapor rose and was condensed by the condenser which was connected to a cold-water source using a hose. The water entered through inlet in the condenser to cool off. The n-hexane vapor condensed back and made contact with the sample again thereby aiding extraction. As it condensed and dropped back on the sample, it rose until it reached a refluxing time and refluxed back into the round bottom flask with oil mixed with it. After it had refluxed for a number of times, the extracted sample was removed and the solvent mixture in the flask was separated by simple distillation and the solvent was recovered for further use.

Extraction and Determination of Percentage Oil

Soxhlet extraction method was used to extract oil from water melon seed. The soxhlet extractor was set up and exactly 20 g of the ground seed sample was weighed out accurately using the analytical weighing balance and dried extraction thimble of known weight (w1) was used to wrap the seed sample. The weight of the thimble together with the sample was taken (w2). The thimble with the sample was dropped into the soxhlet extractor which was fitted into the neck of a 250ml capacity round bottom flask containing boiling chips which controlled bumping. Sufficient quantity of n-hexane was poured into the extractor to wet the sample wrapped in the thimble. Considerable quantity of n-hexane was poured into the round bottom flask. The soxhlet extractor with the thimble was fixed to the neck of the round bottom flask. Then it was clamped to the retort stand and finally the reflux condenser was coupled to the extractor. The heating mantle was placed under the round bottom flask and was switched on. Heating was maintained at a temperature range of 40-60°C. The heating lasted for 24 hours after which the thimble was removed and the n-hexane was recovered by heating the round bottom flask containing mixture of n-hexane and oil. The n-hexane rose up and condensed back into the soxhlet extractor where it remained in the round bottom flask.

Oil Recovery

The remaining solvent (n-hexane) was totally removed by pouring the content of the round bottom flask into a beaker and distilling over a water bath and then the remaining n-hexane evaporated to the air at about 105°C

for 30 minutes. Over 50% of the n-hexane was recovered. The percentage by mass of the oil in the sample was calculated from the difference in weight (w3).

$$\% \text{ oil yield} = \frac{\text{weight of oil}}{\text{weight of sample}} \times 100$$

Acid Value Determination

0.5 g of the sample (oil) was weighed into a conical flask and was dissolved with 20ml of concentrated ethanol. Three drops of phenolphthalein indicator was added and it was titrated with 0.1M of NaOH to a pink end point. The number of milligrams of NaOH required to neutralize 0.5g of the oil was then calculated as Acid Value. Blank titration was carried out at the same time and under the same condition and the volume used (B) recorded.

$$\text{Acid value} = (S-B) \cdot M \cdot \text{molar mass of NaOH}$$

Where S is the standard alkali used for the titration of the sample (ml).

B is the standard alkali used for titration of the blank (ml)

M= Molarity of the standard alkali

W=Weight in g of the sample (oil)

Methods: Dijkstra (2016), AOCS: Cd3a-63j and

DGF: C-V-2

2.5 Free Fatty Acid Determination

Free fatty acid is calculated from acid value.

Free fatty acid= Acid value

Iodine Value Determination

1g of sample (oil) was weighed into a conical flask and 15ml of chloroform was added. Small quantity of acetic acid was added and 25 mls of Wijs solution was also added. The contents of the flask were shaken very well. The resultant solution was kept in a dark cupboard for 2 hours during which it was shaken every thirty minutes. 20 ml of 10% potassium iodide was added to the solution with 120ml of distilled water and it was shaken properly. The colour changed to red and the red solution was titrated with 0.1M of sodium thiosulphate until red colour disappeared from the solution. 5mls of 1% starch indicator was added to the solution and the colour turned to blue black. The solution was titrated again with 0.1M sodium thiosulphate and the colour turned to colourless which was the end point. Blank titration was carried out at the same time and under the same condition and the volume used (Bml) recorded.

$$\text{Iodine value} = M \times (Bml - Sml) \times n$$

Weight of the sample

M = Molarity of sodium thiosulphate

Bml=Blank titer

Sml=Sample titer

n= Constant

W= Weight in g of sample used

Saponification Value Determination

1g of oil was weighed into a conical flask and 50ml of 1M ethanolic sodium hydroxide was poured into the oil. The mixture was refluxed using a round bottom flask containing boiling chips and a 3 drops of phenolphthalein indicator. The round bottom flask was connected to a reflux condenser and was heated for about 45 minutes. The essence of refluxing was to get a perfect dissolution of oil in the ethanolic sodium hydroxide. The solution was titrated against 0.5M HCl so as to get the quantity of caustic soda consumed by fat during saponification. Blank titration

was carried out at the same time and under the same condition and the volume used (Zml) recorded.

Saponification value = $\frac{MX(Zml-Yml)}{W}$ X molar mass of NaOH

Wt of sample used

Where Zml= Titer for blank=Zml of 0.5MHCl

Yml= Titer for the determination=Yml of 0.5M HCl

M= Molarity of the acid

W=Weight in g of the sample used

Specific Gravity Determination

13.950ml specific gravity bottle was weighed and dried and later weighed and the weight (W) was noted. The bottle was filled with distilled water and was weighed again and the weight (W1) was noted. The water was poured out and the specific gravity bottle was dried and filled with the oil and weighed. The weight of the bottle together with the oil (W2) was also noted. The specific gravity was calculated using the formula

Specific gravity = $\frac{W2-W}{W1-W}$ or weight of oil

W1-W weight of water

Where W=weight of specific gravity bottle

W1= weight of bottle with water

W2 = weight of bottle with oil

Formulation of Hair Shampoo from water melon seed

10g of sodium hydroxide pellets was weighed into the beaker and was dissolved with 50ml of distilled water stirring continuously with a glass rod. 20g of water melon seed oil was added and stirring continued. Heating was done for 5 minutes after which it was cooled and 15ml of absolute ethanol (99.8%) was added while the stirring continued. 15ml of formaldehyde was also added and stirring was done for a few minutes. 2 drops of Hand R colour was added and stirring done to get a homogenous solution. Finally, carboxyl methyl cellulose was added as thickener and stirring was done before pouring into a container.

RESULTS AND DISCUSSION

The results of the water melon seed oil are shown in Table 1. The oil extracted from the water melon seed is light yellow and the percentage oil yield show that it can be produced in commercial quantity. The oil is liquid at room temperature which shows that it can be used in the formulation of biodiesel. From Table 3.1, the acid value is low. This shows that the oil is edible. The free fatty acid content is low which indicates that the oil has a long shelf life. The free fatty acid content fell within some range reported by Akanya *et al.*, 1990 for cotton seed extract. The high saponification value shows that the oil will be good for production of soaps, creams and hair shampoo. Saponification is the hydrolysis of esters and the saponification value is the number of milligrams of potassium hydroxide required to saponify 1gram of oil or fat (Jacobs, 1999). The iodine value shows that it is a non-drying oil and the value is comparable with that reported for coconut (Akanya *et al.*, 1990). The quantity of iodine absorbed is a measure of the unsaturation of an oil or fat and the iodine value is generally expressed as the number of grams of iodine absorbed by 100grams of the oil. Drying oils with a high percentage of unsaturated fatty acid for

Table 1: Results of Characterization of Water Melon Seed Oil

Analytical parameters	Values
Oil content (%)	40
Acid value(mg/g)	2.4
Free fatty Acid (mg/g)	1.2
Iodine value (mg/100g)	1.47208
Saponification value (mg/g)	688
Specific gravity (28°C)	0.909

example linseed oil containing 67% unsaturated C18, have iodine value in the range 108-120 (Litchfield, 1982). On exposure to air, drying oils form solids and find useful application in the paint industry. Non-drying oils for example coconut oil with iodine value of 10 contained about 13% palmitic acid and less than 1% of unsaturated acids. Nondrying oils find little use in both human and animal diet. The semi-drying oils are those with iodine values of 90-100 (Litchfield, 1982, Hitchcock and Nichols, 1971). The oils thicken very slowly when exposed to air. Sesame oil, soya bean oil and cotton seed oil have been classified as semi drying oils. (Litchfield, 1982, Hitchcock and Nichols, 1971 and Eckey, 1954). From the cost of materials used in the production of the hair shampoo, the cost of the shampoo is affordable when compared to market price of other shampoos. Characterization of the oil shows that it has some commercial value and the revenue gotten from it can be used for physical development.

REFERENCES

- Morrison RT and RN Boyd, 1997. Organic Chemistry, 5th Edition, Allyn and Bacon Inc. Boston, USA, pp: 1263-1275.
- Ernesto B, 1976. Batch and Continuous Solvent Extraction, Oil Chemist Society, 53: 275-278.
- Van N, 1976. Scientific Encyclopedia, Van Nostrand Reinhold Company, New York, Cincinnati, pp: 994-1054.
- Nwafor JC, 1995. Utilization of Castor Seed Oil for the Formation of Paint, A BSc. Degree Thesis Presented to the Department of Industrial Chemistry, Nnamdi Azikiwe University Awka.
- Jacobs MB, 1999. The Chemical Analysis of Foods and Food Products, CBS Publishers and Distributors, New Delhi, India, pp: 365-400.
- Ibemesi JA, 1992. Vegetable Oils as Industrial Raw Materials, The Nigerian Perspective, Press, Enugu, pp: 20-24.
- Akanya JN, HO Akanya and MB Mohammed, 1990. Fatty Acid Composition of Lipids Extracted from Some Varieties of Nigerian Cotton Seeds, Nig J Tech Res, 2: 19-22.
- Litchfield C, 1972. Analysis of Triacylglycerides, Acad. Press, New York, USA.
- Hitchcock C and BW Nichols, 1971. Plant Lipid Biochemistry, Acad. Press, London, p27a
- Eckey EW, 1954. Vegetable Fats and Oils, Reinhold New York, pp: 75-79.
- Dijkstra AJ, 2016. Vegetable Oils: Composition and Analysis, Encyclopedia of Food and Health.
- Azogu CI, 2010. Laboratory Organic Chemistry, 2nd Edition, Maybinson Book Publishers, New Jersey, USA, pp: 226-231.